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Determination of particle size distribution by gravitational liquid sedimentation methods

Part 1: General principles, requirements and guidance

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National foreword

This British Standard is the UK implementation of ISO 13317-1:2024. It supersedes BS ISO 13317-1:2001, which is withdrawn.

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Second edition
2024-04

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 orientation

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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 document 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 characterization including sieving*, Subcommittee SC 4, *Particle 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:

— core terms and definitions have been revised;

— the explanation of measurement principle and techniques has been revised and expanded;

— 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.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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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 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 can 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 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 the 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 the analysis theory. In general, the volume fraction of particles is well below 1 %.

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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*

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 generally, "rising" or "floating".

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3.2
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 the 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 is used for calculating the Stokes diameter applies also to coated particles or multiconstituent particles (such as droplets in multiple emulsions). The buoyant density 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 close-packed *agglomerates* (3.8) or aggregates, the buoyant density can be replaced by the apparent particle density – with particle referring to the agglomerate or 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 particle 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 26824:2022, 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.

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 with "liquid" in the definition.]

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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 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 of the sample mass and the volume of the sample including the volume of closed pores (if present) but excluding the volume 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 — "as well as that of void spaces between particles within the bulk sample" has been deleted from the definition and Note 1 to 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 surfaces 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 the 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 for porous particles and particle *agglomerates* (3.8) in particular.

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 for more information).

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 and droplets of multiple emulsions) can be approximated with the averaged densities of the single constituents.

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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), particularly 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, the *buoyant* (particle) *density* (3.16) is used, 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 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 applies 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 with a diffusional particle flux that 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 the Lambert-Beer limit), refractive index increment or X-ray attenuation.

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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”

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 depending 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.

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: The limit of quantity detection depends on factors such as size range, precision, noise level and smoothing algorithms.

Note 2 to entry: The limit of quantity detection affects the *lower size limit* (3.21) and *upper size limit* (3.22).

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 apply.

a	edge length	m
Ar	Archimedes number	dimensionless
b	systematic deviation of measured value from true value	varying
C	transformation coefficient, see Formula (34)	$m^{0,5} \cdot s^{0,5}$
C_D	drag coefficient	dimensionless

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$c(x)$	concentration density	varying
c_M	concentration with respect to extensive property M	varying
d	diameter	m
D_p	particle diffusion coefficient	$m^2 \cdot s^{-1}$
F_D	drag force (also: hydrodynamic resistance)	N
g	gravitational acceleration	$m \cdot s^{-2}$
h_{sed}	sedimentation distance	m
k	coverage factor	dimensionless
k_B	Boltzmann constant	$J \cdot K^{-1}$
L	length	m
Lj	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	resolution ratio	dimensionless
Pe	Péclet number	dimensionless
Q_{toq}	cumulative function of a distributed quantity, for a type of quantity, in which the fractions are weighted	dimensionless
q_{toq}	density function of a distributed quantity, for a 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	$m \cdot s^{-1}$
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

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$\Delta\rho$	density contrast	$kg \cdot m^{-3}$
$\dot{\gamma}$	shear rate	s^{-1}
δ_{layer}	layer thickness	m
δ_x	relative error of the calculated particle size	dimensionless
ε	porosity	dimensionless
η_c	viscosity of the continuous phase	$Pa \cdot s$
ρ_p	particle density	$kg \cdot m^{-3}$
ρ_c	density of the continuous phase	$kg \cdot m^{-3}$
ρ_d	true density of the dispersed phase	$kg \cdot m^{-3}$
φ_V	volume fraction	dimensionless

For the purposes of this document, the following subscripts apply.

aggl	agglomerate, also aggregate
app	apparent
bouy	buoyant
c	combined
incl	inclusion
lab	laboratory
max	maximum
occl	occluded voids
open	open pores
ref	reference
rel	relative
rep	repeatability
Rw	reproducibility
sk	skeleton
sus	suspension
toq	type of quantity
tot	total

For the purposes of this document, the following abbreviated terms apply.

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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

OV occluded void

PTA particle tracking analysis

QCM quality control material

RM reference material

SOP standard operating procedure

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 [43], 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 document (i.e. for creeping flow). It amounts to 0,2 µs and 49 ms for spherical gold particles (r_p = 19 300 kg/m^3) of 0,3 µm and 100 µm, 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.

Sedimentation results in a depletion of the dispersed phase and the formation of a (fixed) layer of separated particles – either at the bottom (sediment) or at the air-liquid interface (cream, foam).

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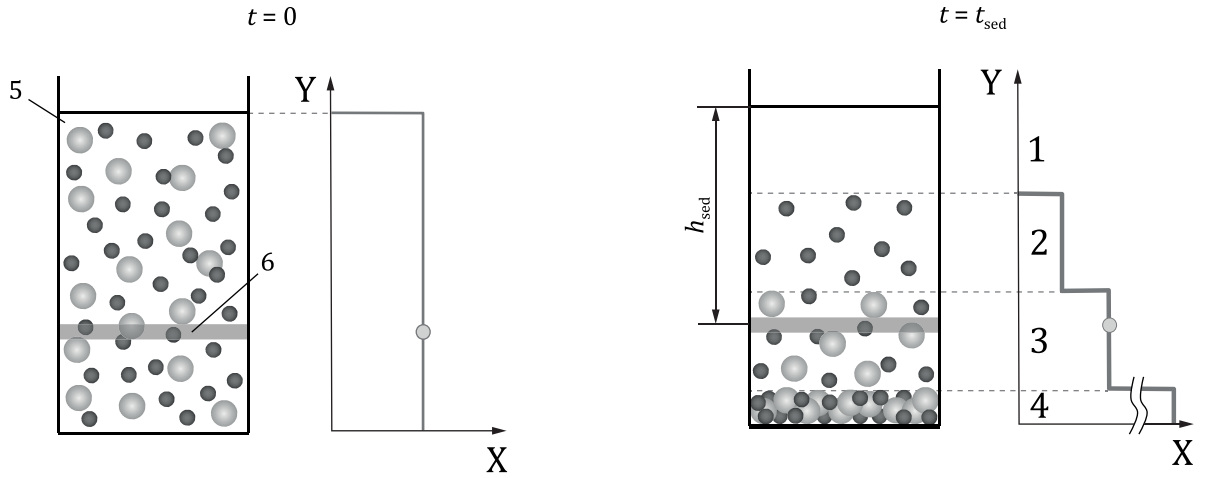

a) Homogeneous dispersion and corresponding concentration profile at $t = 0$

b) Formation of four zones and corresponding concentration profile at t_{sed}

Key

X concentration
Y position
1 zone 1 = particle-free supernatant
2 zone 2 = depleted dispersion (due to loss of coarse particles)
3 zone 3 = original dispersion
4 zone 4 = sediment
5 sedimentation cell
6 measurement zone
h_{sed} sedimentation distance
t_{sed} sedimentation time

Figure 1 — Schematic illustration of phase separation due to sedimentation for a bidisperse sample with positive density contrast and its monitoring by an incremental sensing technique

Particles settle at different velocities in the dilute regime based on their difference in size. As a result, four particle concentration zones are progressing (see Figure 1). The top layer, the supernatant (zone 1), has already become free of particles ($c = 0$). The next layer (zone 2) is depleted of the large particles and the dispersed phase consists only of the fine particle fraction ($c = c_{fine}$). Simultaneously, a sediment (zone 4) develops at the bottom. In the early stages of phase separation, it is predominantly built up by fast settling, coarse particles. In the layer just above the sediment (zone 3), the concentration does not change at all with time ($c = c_{initial} = c_{fine} + c_{coarse}$) in the case of gravity sedimentation until the coarse fraction has completely entered the sediment (zone 4). The sedimentation process is finished when all particles have settled down and the formation of the sediment is completed. In the case of a negative excess force, the opposite picture is observed, i.e. particle concentration decreases at the bottom and increases at the top.

In general, sedimentation techniques quantify either the changes of local particle concentration in the measurement zone (detection level) or the growth of the layer of separated particles (e.g. at the bottom of a detection tray for the balance method, see ISO 13317-4). The vertical position of measurement zone (see Figure 1) and the time elapsed since starting the separation define the sedimentation velocity (first measurement principle). Further preconditions or assumptions are not needed for attributing the sedimentation velocity to a point of the measurement data (e.g. be it the temporal evolution of local particle concentration, the local variation of particle concentration at a given time, the time function of sediment weight). The sedimentation velocity itself constitutes an essential characteristic for several applications,

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which goes far beyond a granulometric characterisation (e.g. analysis of suspension stability, segregation phenomena, flocculation processes and phase separation of highly concentrated slurries).

The sedimentation velocity depends on the properties of the continuous phase (density, rheological behaviour) and on characteristics of the particles (i.e. size, density and shape). This allows its conversion into particle size based on the Stokes' law and corresponding presumptions (see 6.2.1). This (indirect) quantification yields an equivalent diameter with respect to sedimentation velocity, called Stokes diameter, x_{Stokes}. It reflects the weight or volume of the particles as well as their mobility or drag coefficient. It is typically one of the smallest equivalent diameters of a given particle. According to Leschonski[44], Formula (1) applies:

$$x_{\mathrm{Stokes}} < x_V < x_{\mathrm{S}} = x_{\mathrm{proj,m}} < x_{\mathrm{proj,st}} \tag{1}$$

where

$x_{\mathrm{proj,m}}$ is the projected area equivalent diameter for mean orientation;

$x_{\mathrm{proj,st}}$ is the projected area equivalent diameter for stable orientation;

x_{S} is the surface equivalent diameter;

x_V is the volume equivalent diameter.

The difference between these equivalent diameters is particularly pronounced for particle aggregates and agglomerates, where Formula (2) applies[45]:

$$x_{\mathrm{Stokes}} << x_{\mathrm{hd}} < x_{\mathrm{gyr}} < x_{\mathrm{Feret,max}} \tag{2}$$

where

$x_{\mathrm{Feret,max}}$ is the maximum Feret diameter;

x_{gyr} is the diameter of gyration;

x_{hd} is the hydrodynamic equivalent diameter.

NOTE 2 The Stokes diameter is not identical to the hydrodynamic or mobility equivalent diameter, which solely refers to the particles drag coefficient. Colloid scientists often use the radius of gyration (i.e. $0{,}5 \cdot x_{\mathrm{gyr}}$).

An essential feature of each sedimentation technique is the way of quantifying the particles and thus particle size fractions. It occurs separately from the physical process of particle classification via sedimentation and can employ very different measurands (e.g. local mass concentration, local X-ray attenuation or light obscuration, sediment weight, sediment height). The primary measurement results of sedimentation techniques are typically time-curves of these measurands and sometimes they can also be the distribution of these measurands along the vertical coordinate (i.e. profiles). The positions at which time-curves are measured or the instants at which a profile is acquired can be adjusted by operators or are fixed by instrumentation. The time axis or the vertical measurement positions can be easily transformed into an axis of sedimentation velocity on which the measured quantity is projected. Hence, the above-mentioned measurands define the intrinsic type of quantity, by which the individual size fractions are weighted (see 5.2 and Annex A). The conversion of these quantities to fundamental ones (i.e. volume or number) can rely on an unbiased or biased linear scaling or can involve parametric models (e.g. for light obscuration).

5.2 Technical realisation of sedimentation-based measurement techniques

There is a considerable variety of analytical techniques, which characterise particle systems based on the sedimentation velocity (or equivalently the Stokes diameter). In general, they monitor the sedimentation induced separation of the disperse sample via the depletion of the dispersion or the growth of deposited particle layer (sediment or cream). Yet, they differ in several aspects.

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At first, fundamental difference is the driving force. This document only covers sedimentation under gravity, while the techniques based on sedimentation in centrifugal fields are described within the ISO 13318 series. A further distinction refers to the type of the primary measurand: some techniques probe integral properties of the segregated particles (e.g. sediment mass or volume), others address an integral (cumulated) quantity of dispersed particles (e.g. via hydrostatic pressure) and a third group measures the local particle concentration within the dispersion (e.g. via X-ray attenuation or light extinction). With regard to data analysis, the two former types of measurement techniques are called integral (or cumulative) techniques, whereas the latter types are called incremental techniques (see Figure 2).

Integral sedimentation techniques comprise all techniques that monitor the growth of the sediment, such as sedimentation balances techniques (see ISO 13317-4) or sedimentations tubes (see ISO 6344-3 and ISO 8486-2). These techniques apply to all disperse systems of solid particles with a positive density contrast to the liquid phase. They measure mass, volume or height of the sediment as a function of time. Another group of integral techniques probe the total amount of (solid) particles, which is still suspended in the continuous phase, for which purpose they, for example, measure the hydrostatic pressure (manometric sedimentation techniques[46]) or the weight force of a diving body (diving balance techniques[47],[48]).

Incremental sedimentation techniques measure the local particle concentration. One way to accomplish this consists of periodic sampling with a pipette and subsequent quantification of dispersed material (pipette techniques, see ISO 13317-2). Alternatively, these techniques probe local, macroscopic properties of the sample, for example, dispersion density (with micro-diving bodies, i.e. small hydrometers, see ISO 17892-4, ISO 11277 and ASTM D7928-17), X-ray attenuation (X-ray sedimentometer, see ISO 13317-3), light extinction or scattering (photosedimentometer, see GB/T 6524, ANSI B74.20) or refractive index[49]. Pipette-based and hydrometric sedimentation techniques were rather popular in the past, whereas modern sedimentometers typically employ radiation-based sensing technologies, especially for fine particles $\ll 100$ µm.

Figure 2 illustrates the differences between integral and incremental sedimentation techniques through examples of monitoring the growth of sediment (integral) and the continuous observation of local particle concentration (incremental). Time curves of the corresponding measurement signals are shown for two modes of operation:

a) HSM (also suspension mode, situation A), and

b) LSM (situation B).

The former, which is preferably used in gravitational sedimentation analysis, starts with thoroughly mixed, homogeneous samples, whereas in case of the latter, the initial state is characterised by a thin layer of dispersion sample on top of a particle-free liquid. Although, the line-start method is prone to adverse hydrodynamic instabilities (e.g. density convections), it is still used for specific applications (e.g. ISO 6344-3) because it offers a few advantages such as the determination of the coarsest grain size. The two different operational modes coincide with different shapes of time-curves (see Figure 2) and require different procedures in data analysis (see 6.2.2).

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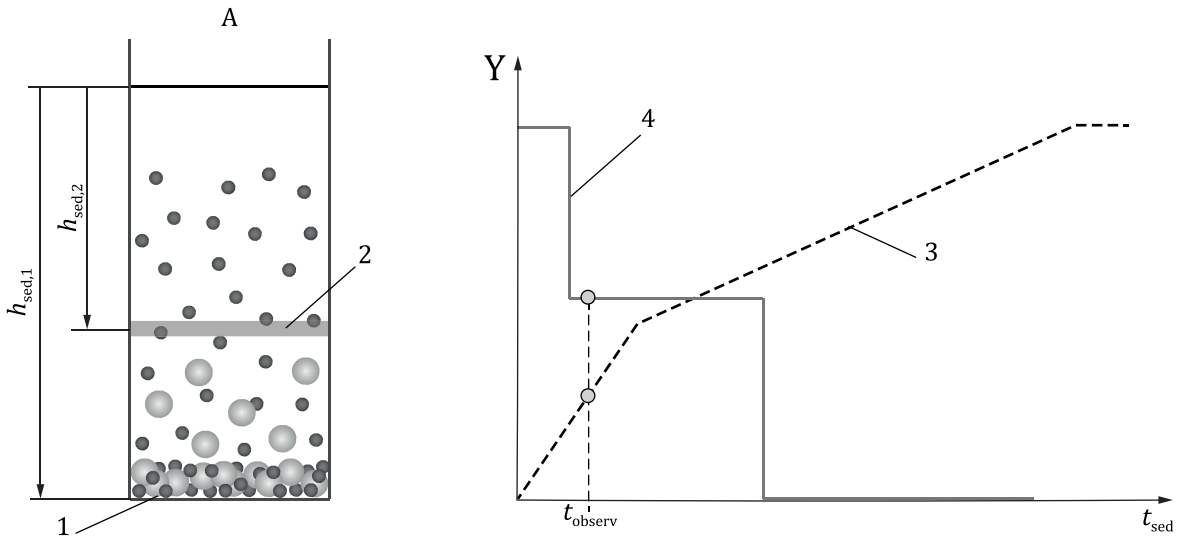
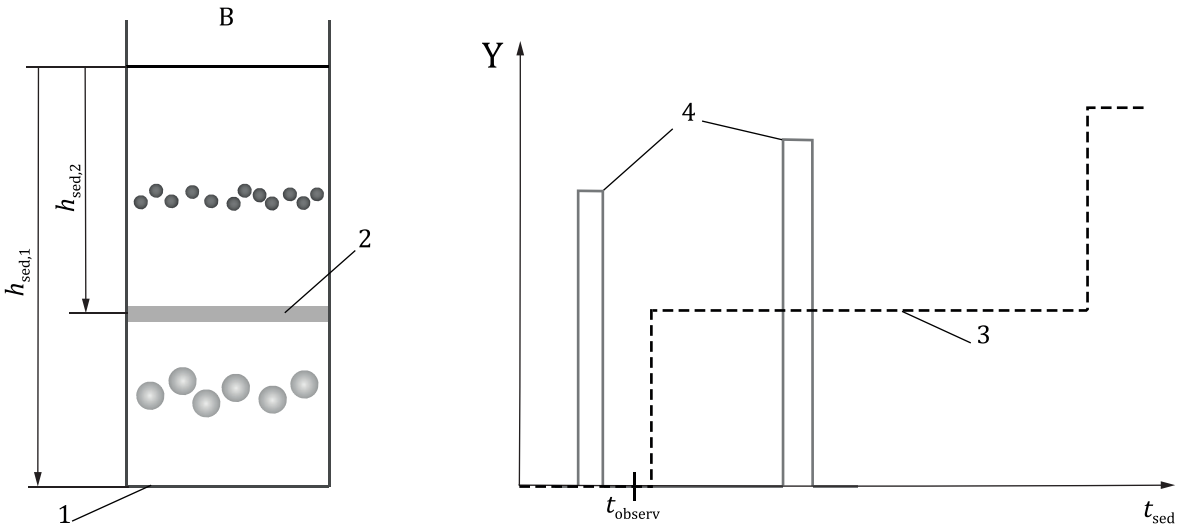


a) Sedimentation for HSM used for incremental techniques and integral techniques



b) Sedimentation for LSM used for incremental techniques and integral techniques

Key

t_{sed}	time elapsed since starting the sedimentation experiment
Y	observed quantity (e.g. sediment mass or X-ray attenuation)
1	sediment layer [an example of measured object for integral (cumulative) techniques]
2	measurement zone for incremental (differential) techniques
3	time curve for sediment quantity (e.g. mass or volume)
4	time curve for local particle concentration at measurement zone
$h_{sed,1}$	sedimentation distance for 1, from which the sedimentation velocity at t_{sed} is calculated
$h_{sed,2}$	sedimentation distance for 2, from which the sedimentation velocity at t_{sed} is calculated
t_{observ}	time point of observation for situations on left-hand side

Figure 2 — Example of sedimentation growth monitoring using gravitational sedimentation techniques and corresponding time curves of observed quantities

Incremental techniques use different modes of sensing. Traditionally, the measurements are conducted at a fixed vertical position (see Figure 1) or are gradually changed over a vertical range (scanning mode, (see Reference [43], chapters 9.4.4 and 9.5]). The sedimentation distance refers either to the liquid surface (meniscus) for positive density contrast or to the bottom for a negative one. A recently introduced

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sensing technology realises a time-resolved measurement of the complete concentration profile (STEP-Technology®[1)][50]), which considerably enhances the data analysis (see ISO 13318-2) and e.g. facilitate the distinction between settling and creaming phases.

Despite the significant differences among the various sedimentation techniques, they all directly measure the same particle property, the settling velocity or Stokes diameter and should therefore yield identical results for monodisperse particles of arbitrary shape. However, the instrument's response to polydisperse samples can substantially scatter because the various measurands (e.g. sediment height, X-ray attenuation, light scattering) are differently affected by the individual size fractions. Indeed, the experimental method or sensing technique employed for quantifying the amount of particles with a certain sedimentation velocity determine the intrinsic type of quantity, in which the originally measured distribution functions are weighted.

Annex A lists main characteristics and references for selected sedimentation techniques.

6 Measurement data and basic rules of data evaluation

6.1 Sedimentation velocity distribution

The primary result of sedimentation techniques is the distribution of the sedimentation velocity. In general, the observed sedimentation velocity is a collective property, which depends on particle interactions and the particle concentration, which allows for an evaluation of typical quality endpoints such as phase separation or flocculation. However, within the scope of this document (i.e. dilute particle systems), the sedimentation velocity constitutes a dispersity property, i.e. an individual property of the respective particles. It provides information about the particles that goes beyond particle size. For instance, it can be used to distinguish between creaming and settling constituents in complex formulations with multiple dispersed phases. Last but not least, velocity distributions allow an evaluation of polydispersity for particulate materials of unknown composition or in the event of lacking data needed for the transformation of sedimentation velocity into particle size.

Sedimentation techniques relate a property of the dispersion phase (zones 1 to 3 in Figure 1) or the sediment (zone 4 in Figure 1) to the time elapsed t_{sed} since starting the gravitational phase separation in a thoroughly mixed sample. Such properties are the local particle concentration, the total mass of dispersed matter above a probe or the height of a sediment layer. They are measured at a certain vertical position below the liquid surface, can be thus related to the maximum settling distance h_{sed} for all particles, which pass the measurement position at t_{sed} (see Figure 2). Elapsed time and settling distance define the sedimentation velocity as shown in Formula (3):

$$v_{\mathrm{sed}} = \frac{h_{\mathrm{sed}}}{t_{\mathrm{sed}}} \tag{3}$$

NOTE For creaming or rising particles, the relevant travelling distance h_{sed} is defined as the vertical distance from the bottom of the vessel. In this case, a negative sign is assigned to the velocity.

The distribution of the sedimentation velocity can be described in analogy to particle size distributions, yet the possible occurrence of negative sedimentation velocities (i.e. counter-directed to gravitational acceleration) requires the pragmatic convention of only considering their absolute values:

$$Q_{\mathrm{toq}}(v) = \int_0^v q_{\mathrm{toq}}(v_{\mathrm{sed}})\,\mathrm{d}|v_{\mathrm{sed}}| \tag{4}$$

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$$q_{\mathrm{toq}}(v) = \left.\frac{\mathrm{d}Q_{\mathrm{toq}}(v_{\mathrm{sed}})}{|\mathrm{d}v_{\mathrm{sed}}|}\right|_{v} \tag{5}$$

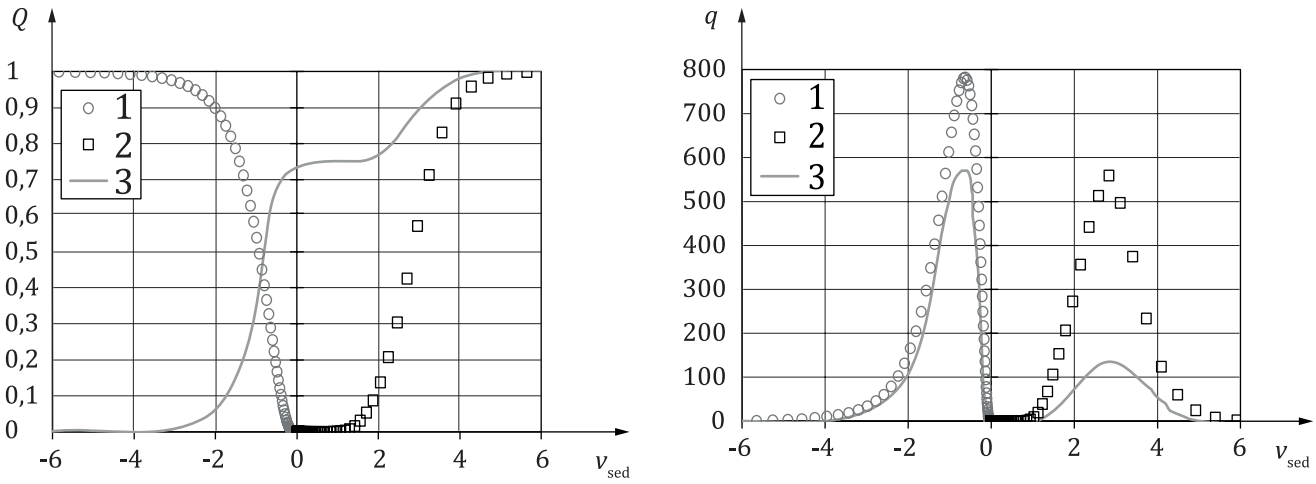
The functions Q_{toq} and q_{toq} are the cumulative and density function of the velocity distribution. The index "toq" denotes the type of quantity, in which the "amount of dispersed phase" is quantified. The introduction of the absolute values (| |) ensures that, for creaming particles, the cumulative function rises from zero at $v_{\mathrm{sed}} = 0$ to one at $v_{\mathrm{sed}} \to -\infty$. This typically coincides with mirroring the cumulative and density functions of creaming particles at the ordinate. However, if creaming and settling occurs simultaneously, Formulae (4) and (5) need to be replaced by expressions, that allow a variation in the sign of the velocity values:

$$Q_{\mathrm{toq}}(v) = \int_{-\infty}^{v} q_{\mathrm{toq}}(v_{\mathrm{sed}})\,\mathrm{d}v_{\mathrm{sed}} \tag{6}$$

$$q_{\mathrm{toq}}(v) = \left.\frac{\mathrm{d}Q_{\mathrm{toq}}(v_{\mathrm{sed}})}{\mathrm{d}v_{\mathrm{sed}}}\right|_{v} \tag{7}$$

These conventions are illustrated in Figure 3 for the sedimentation velocity of oil droplets and polystyrene particles in aqueous solution (for each material separately and for a mixture of both). While the polystyrene particles settle down to the bottom of the vessel, oil droplets rise up, i.e. they move against the direction of gravitational acceleration with a negative sedimentation velocity.

Velocity distributions can be similarly used as particle size distributions; modal values $v_{\mathrm{mod,toq}}$ localise the most frequent velocities and quantiles $v_{P,\mathrm{toq}}$ correspond to a value P (in percent) of the cumulative function. For instance, the main quartiles of the velocity distribution were successfully employed to quantify the polydispersity by the "sharpness" (which can be quantified by the ratio $v_{25,\mathrm{toq}}/v_{75,\mathrm{toq}}$) of the sedimentation front of concentrated suspensions[51]. Furthermore, velocity distributions indicate directly polydispersity and allow to rate the effectiveness of disruptive processing (see ISO/TS 22107).



a) Cumulative functions **b) Distribution density**

Key

v_{sed} sedimentation velocity, in µm/s

Q cumulative function (extinction-weighted) of the velocity distribution

q density function (extinction-weighted) of the velocity distribution, in s/mm

1 oil-in-water emulsion (ρ_{oil} = 917 kg/m^3, $x_{50,3}$ = 7 µm)

2 polystyrene suspension (ρ_{PS} = 1 055 kg/m^3, $x_{50,3}$ = 9 µm)

3 mixture

Figure 3 — Sedimentation velocity distributions of an oil-in-water emulsion (negative velocities), a polystyrene suspension (positive velocities) and a mixture of both (1:1 by weight, continuous line)

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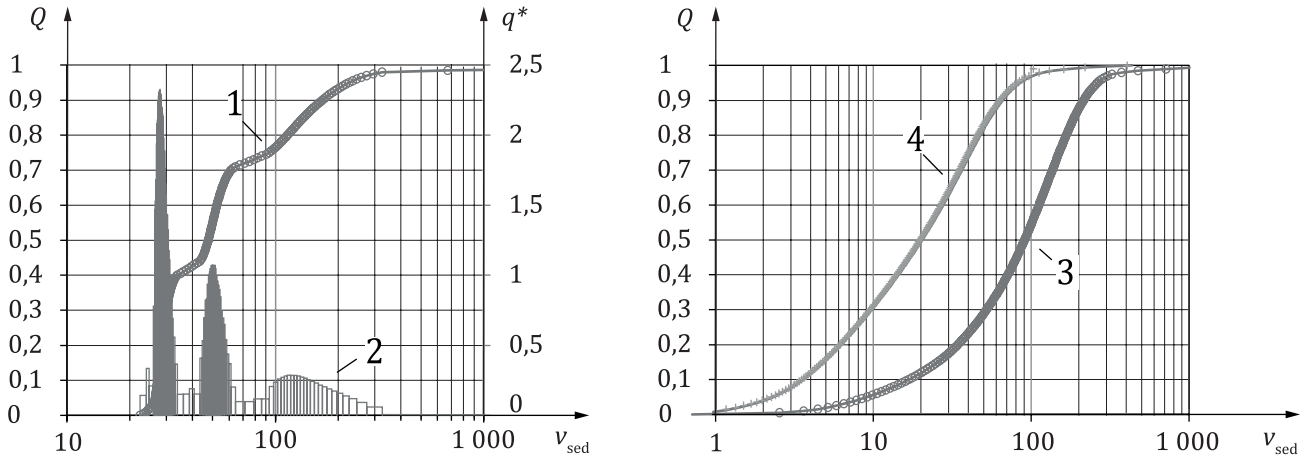
Distributions of the sedimentation velocity reflect the state of dispersion (at least for sufficiently dilute samples). That means they can be used to assess polydispersity or multimodality [see Figure 4 a)], the presence of coarse contaminants or the dispersibility of a particulate material [see Figure 4 b)]. Their main advantage is that they do not require detailed knowledge of the material properties of the continuous and dispersed phases. Direction of particle motion for sedimenting or creaming particles results in "velocities" of different sign (falling is equivalent to positive velocity, creaming is equivalent to negative velocity) as shown in Figure 3 and allows an easy classification.

Velocity distributions are plotted for the intrinsic type of quantity, by which the different particle fractions contribute to originally measured properties of the partly demixed dispersion or the separated particles (i.e. sediment or cream layer). For instance, X-ray and photometric transmission measurements of local particle concentrations lead to X-ray-absorption-weighted and (light) extinction-weighted distributions, respectively. A direct conversion with respect to the type of quantity is typically not possible because such a conversion relies on the relationship between quantity and particle size (see 6.2.2.6).

The disadvantage of sedimentation velocity is its dependency on the properties of the continuous phase (density, viscosity), which restricts its applicability and impedes its transferability to arbitrary situation of sedimentation-induced particle separation. Distributions of the sedimentation velocity are therefore transformed to ones of particle size (see 6.2) for most analytical purposes.



a) Velocity distribution of a multimodal suspension of PMMA particles

b) Velocity distribution of a silica suspension due to different dispersive processing

Key

v_{sed}	sedimentation velocity, in µm/min
Q	cumulative function (extinction-weighted) of the velocity distribution
q^*	transformed density function (extinction-weighted) of the velocity distribution
1	cumulative function
2	transformed density function (histogram)
3	feed
4	after milling

Figure 4 — Application examples of sedimentation velocity distribution

For the sake of completeness, it should be mentioned that sedimentation velocity is often replaced by the sedimentation coefficient in centrifugal sedimentation – especially in the ultracentrifugation community. This coefficient relates the particle velocity to the acceleration of the applied force field (i.e. gravitational or centrifugal acceleration) and is expressed in units of time (see ISO 13318-1). Unlike with sedimentation velocity data, sedimentation coefficient data facilitate a direct comparison of results from gravitational and centrifugal sedimentation.

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6.2 Stokes-based analysis for obtaining particle size distributions

6.2.1 Particle size

The sedimentation of particles is induced by gravity and retarded by hydrodynamic resistance. The terminal sedimentation velocity is thus affected by their volume and hydrodynamic mobility. Both parameters are size-dependent which allows to calculate the particle size from the sedimentation velocity [see Formula (3)].

For small spherical particles of diameter *x*, Formula (8) applies[52]:

$$v_{\mathrm{sed}} = \frac{g\left(\rho_{\mathrm{p}} - \rho_{\mathrm{c}}\right)}{18\eta_{\mathrm{c}}} x^2 \tag{8}$$

where

g is the gravitational acceleration;

η_{c} is the dynamic viscosity of the continuous phase;

ρ_{c} is the density of the continuous phase;

ρ_{p} is the density of the particle.

The validity of Formula (8) relies on a series of assumptions, which are referred to as “standard theory” and comprised of the following.

a) The motion is slow, which means that creeping flow conditions applies (i.e. Stokes flow) and the hydrodynamic drag obeys Stokes' law for spherical objects (see also Clause C.1).

b) There are only three forces that act on the particles: gravity, buoyancy and viscous drag (see also Clause C.2).

c) Gravity and buoyancy can be calculated according to Newton’s law and Archimedes’ principle.

d) The liquid is incompressible and Newtonian (see also Clause C.3).

e) A particle’s sedimentation is not affected by the presence of other particles (see also Clause C.4).

f) The sphere is isotropic and has a smooth and rigid surface (see also Clauses C.5 and C.7).

g) The sedimentation is not affected by the walls of the vessel (see also Clause C.8).

h) The terminal sedimentation velocity is achieved instantaneously (see also Clause C.9).

i) The particles do not experience changes during the experiment (e.g. no dissolution, no swelling, no agglomeration).

Last but not least, the gravitational acceleration, *g*, is commonly regarded as a physical constant, but it is not. The standard value of 9,806 65 m/s^2 applies approximately to a latitude of 45°, whereas the equatorial value is 9,780 m/s^2 and the polar value amounts to 9,832 m/s^2 (i.e. the maximum deviation from the standard value is <0,3 %).

In practice, it is rather unlikely that the particles are spherical and that all assumptions listed above apply. In particular, particles of most real-world materials are commonly neither spherically shaped nor non-agglomerated. For that reason, one conventionally accepts the invalidity of all assumptions concerning the particulate phase, as long as the remaining assumptions are met by adequate sample preparation and proper measurement conditions. Measured sedimentation velocities are then transformed into particle size

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