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Determination of particle size distribution by gravitational liquid sedimentation methods

Part 5: Photosedimentation techniques

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The UK participation in its preparation was entrusted to Technical Committee LBI/37, Particle characterization including sieving.

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Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 5: Photosedimentation techniques

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

Partie 5: Techniques de photosédimentation

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Foreword

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

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Introduction

The principles of gravitational photosedimentation and its potential use for the granulometric characterization of particle systems have been known for several decades. Recent developments in optoelectronics and data processing have boosted the commercial success and popularity of this measurement technique, which is currently employed in manifold academic and industrial applications.

This document is a part of the ISO 13317 series that provides a general overview on the principles, techniques, methods and underlying physics of particle size analysis by gravitational sedimentation. Photosedimentation employs photometric signals (i.e. transmitted, reflected or scattered light) in order to monitor the changes in the local particle concentration, which arise by the downward or upward particle migration under gravity (called hereafter sedimentation). The temporal or spatial functions of these signals can be directly transformed to distributions of the sedimentation velocity, without referring to model assumptions or being restricted by essential preconditions. Provided the applicability of Stokes' law on particle mobility, one can derive equivalent diameters from the sedimentation velocities (the *Stokes diameter*) and the corresponding particle size distributions. Size fractions are then intrinsically weighted by photometric quantities (e.g. light extinction or scattered light intensity), which is in contrast to the sedimentation techniques described in ISO 13317-2, ISO 13317-3 and ISO 13317-4. However, conversion into volume-weighted distributions is often an integrated part of signal processing, which employs established models for light-particle interactions. A noteworthy feature of gravitational photosedimentation is its ability to finely resolve details in the particle distribution functions. This is related to the physical fractionating of particle systems under gravity and constitutes an advantage compared to spectroscopic ensemble techniques.

Gravitational photosedimentation facilitates the granulometric characterization of dispersed materials of non-zero density contrast to the continuous phase, including solid particles and emulsion droplets. The available measurement range depends on dispersed and continuous phase properties and typically amounts to 200 nm to 100 µm for aqueous samples, whereas the sedimentation velocity can be quantified for the range 0,6 µm/s to 10 mm/s. Also, the working range with regard to particle concentration is strongly affected by material properties and by particle size, yet it is typically well below 1 vol%. The data analysis relies on the assumption that all particles have the same density and comparable shape and do not undergo chemical or physical change in the continuous phase.

In addition, photosedimentation techniques that monitor gravity-induced concentration changes along the complete sample height, e.g. by position-scanning or time-resolved projection, facilitate the characterization of dense dispersion beyond particle size, e.g. with respect to clarification, segregation, agglomeration, consolidation and physico-chemical stability (see ISO/TR 13097). Gravitational photosedimentation is equally applicable in determining particle density (see ISO 18747 series) as well as the formation of sediments and cream layers.

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Determination of particle size distribution by gravitational liquid sedimentation methods —

Part 5: Photosedimentation techniques

1 Scope

This document specifies principles and methods for the use of gravitational photosedimentation techniques for the characterization of dispersed phases of suspensions and emulsions. These techniques monitor the gravity-induced phase separation of particulate materials dispersed in liquids by recording photometric signals (i.e. intensity of transmitted or scattered light) as a function of either vertical position or measurement time, or both.

This document does not cover particle migration by centrifugal, electric or magnetic forces, or sedimentation at high particle concentrations (e.g. zone sedimentation). Moreover, it does not cover the determination of properties other than sedimentation velocity and particle size (i.e. it does not cover particle concentration, particle shape, particle density, zeta-potential or apparent viscosity).

Additionally, this document does not cover alternative techniques for gravitational sedimentation including balance based and X-ray based techniques.

NOTE This document does not purport to address all the safety problems associated with its use.

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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[SOURCE: ISO 13317-1:2024, 3.1]

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).

[SOURCE: ISO 13317-1:2024, 3.2]

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

[SOURCE: ISO 13317-1:2024, 3.3]

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 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 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.

[SOURCE: ISO 13317-1:2024, 3.4]

3.5
shape correction factor
ratio of the sedimentation velocity of a non-spherical particle to the one of a spherical particle of the same volume and *apparent density* (3.15)

[SOURCE: ISO 13317-1:2024, 3.5]

3.6
hindrance function
ratio of the *terminal sedimentation velocity* (3.3) of a *particle* (3.7) placed in well-mixed dispersion divided by its sedimentation velocity in an infinite vessel for the absence of other particles

[SOURCE: ISO 13317-1:2024, 3.6]

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.

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

[SOURCE: ISO 13317-1:2024, 3.8]

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.]

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

[SOURCE: ISO 13317-1:2024, 3.11]

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

[SOURCE: ISO 13317-1:2024, 3.12]

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

[SOURCE: ISO 13317-1:2024, 3.13]

3.14
skeleton density
ratio between sample mass and the volume of the sample including the volume of *closed pores* (3.10) (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 13317-1:2024, 3.14]

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 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.

[SOURCE: ISO 13317-1:2024, 3.15]

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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 surfaces 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, droplets of multiple emulsions) can be approximated with the averaged skeleton 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), particularly for porous particles and particle *agglomerates* (3.8).

[SOURCE: ISO 13317-1:2024, 3.16]

3.17
density contrast
difference between the particle 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.

[SOURCE: ISO 13317-1:2024, 3.17]

3.18
particle Reynolds number
dimensionless parameter expressing the ratio of inertial to viscous forces within a fluid flowing past a particle

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.

[SOURCE: ISO 13317-1:2024, 3.18]

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 applies if the *particle Reynolds number* (3.18) is well below 0,25.

[SOURCE: ISO 13317-1:2024, 3.19]

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.

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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.

[SOURCE: ISO 13317-1:2024, 3.20]

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.

[SOURCE: ISO 13317-1:2024, 3.21]

3.22
upper size limit
size of the largest particle that satisfies the condition of *creeping flow* (3.19) and of which the *terminal sedimentation velocity* (3.3) is detectable

[SOURCE: ISO 13317-1:2024, 3.22]

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 13317-1:2024, 3.23]

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.

[SOURCE: ISO 13317-1:2024, 3.24]

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3.25
limit of quantity detection
smallest quantity of specified particle 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).

[SOURCE: ISO 13317-1:2024, 3.25]

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 Guide 98-3:2008, 2.2.3, modified — Notes 1 to 3 to entry have been deleted and the term "measurement uncertainty" has been added.]

4 Symbols and abbreviated terms

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

Ar	Archimedes number	dimensionless
b	systematic deviation of measured value from true value	varying
C	transformation coefficient, see Formula (27)	$m^{0,5} \cdot s^{0,5}$
C_{ext}	extinction cross section	
c_M	concentration with respect to extensive property M	varying
D_p	particle diffusion coefficient	$m^2 \cdot s^{-1}$
E	extinction	
g	gravitational acceleration	$m \cdot s^{-2}$
h_{sed}	sedimentation distance	m
I	light intensity	
k	coverage factor	dimensionless
K_{ext}	extinction efficiency	
k_B	Boltzmann constant	$J \cdot K^{-1}$
L	optical pathlength	
Lj	Ljaščenko number	dimensionless
M	extensive property indicating the amount of dispersed phase	varying
m	number of bias determinations	dimensionless
N	number of particles	dimensionless

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n	number of replicate analyses	dimensionless
Pe	Péclet number	dimensionless
Q_{toq}	cumulative function of distributed quantity, index “toq” indicates the type of quantity, in which the fractions are weighted	dimensionless
q_{toq}	density function of distributed quantity, index “toq” indicates the type of quantity, in which the fractions are weighted	varying
Re_{p}	particle Reynolds number	dimensionless
s	standard deviation	varying
T	absolute temperature	K
t	time	s
t_{observ}	time point of observation	s
t_{sed}	sedimentation time	s
U	expanded uncertainty	varying
u	uncertainty	varying
V_{meas}	measurement volume	
v_{sed}	terminal sedimentation velocity	$\mathrm{m \cdot s^{-1}}$
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
$\Delta\rho$	density contrast	$\mathrm{kg \cdot m^{-3}}$
δ	thickness	m
η_{c}	viscosity of the continuous phase	Pa·s
ρ_{p}	particle density	$\mathrm{kg \cdot m^{-3}}$
ρ_{c}	density of the continuous phase	$\mathrm{kg \cdot m^{-3}}$
φ_V	volume fraction	dimensionless

In addition, the following subindices are frequently employed.

app	apparent
c	combined
cr	critical
ext	extinction
int	intensity

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lab laboratory

max maximum

meas measurement

psca partial scattering

ref reference

rel relative

rep repeatability

sca scattering

Rw reproducibility

toq type of quantity

Moreover, this document uses the following abbreviated terms.

CRM certified reference material

ILC interlaboratory comparison

NIR near infrared radiation

QCM quality control material

RM reference material

RTM representative test material

UVA ultraviolet A radiation

5 Measurement principle and instrumentation

5.1 General measurement principle

Gravitational photosedimentation allows the characterization of liquid disperse systems based on their phase separation under gravity. Unlike other sedimentation techniques, the phase separation is monitored photometrically as the depletion or accumulation of particles at (a) defined vertical position(s) in the initially well-mixed sample.[1] The measurement can be conducted at a fixed position or a continuously varying one (scanning mode) or even at multiple positions for the same measurement time, including the spatially resolved measurement along the vertical axis. The observed quantity is the intensity of light transmitted through, scattered by, or reflected at the sample. These quantities correlate with the local particle concentration at the measurement position(s). Temporal and spatial changes in local particle concentration are entirely attributed to gravitational sedimentation of the particles. The direction of sedimentary motion depends on the density contrast ($\Delta\rho$) between the dispersed and continuous phase (see 3.17); the corresponding particle motion is either called settling and falling ($\Delta\rho > 0$) or creaming, rising and floating ($\Delta\rho < 0$).

NOTE This document refers to photometric measurement techniques that monitor the gradual depletion or accumulation of particles in the continuous phase, but not to techniques that observe the growth of sediment or cream layers by photometric means (see ISO 6344-3 and ISO 8486-2). Moreover, the operational method requires an initially homogeneous dispersion sample [so called homogeneous-start mode (HSM)]; any approach starting with a thin dispersion layer on top or beneath a particle-free liquid (so called line-start mode) is beyond the applicability of this document.

Figure 1 illustrates the measurement principle for the photometric monitoring of transmitted light (for a scattering configuration, see Figure 3) The example assumes a bi-disperse particle system having a positive

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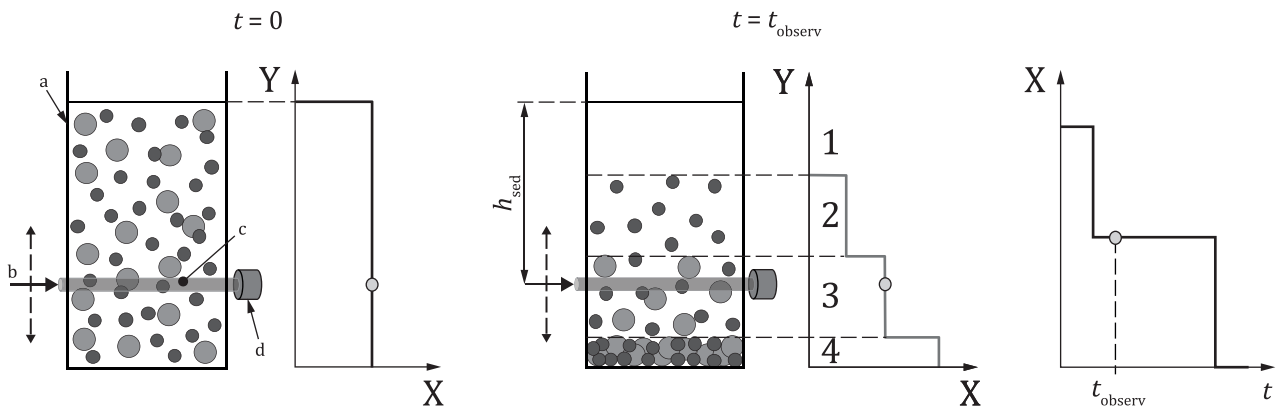
density contrast. At the beginning of the sedimentation analysis ($t = 0$), the sample is well-mixed, i.e. particles of each size fractions are uniformly distributed along the vertical axis and the local particle concentration is independent from position. However, gravity makes the particle settle downward with a size-dependent velocity. This gives rise to four layers differing in particle concentration. A particle-free liquid layer at the top (zone 1). In zone 2, the coarse particles settled out (segregation) within the time $t_{observe}$ and only the fine particles remain ($c = c_{fine}$). The particle content in the third layer does not change at all and composition and concentration are not position dependent and remain at its initial value ($c = c_{initial} = c_{fine} + c_{coarse}$). The sediments forms at the bottom (zone 4), where particles have a very high concentration. Composition is not uniform. The sedimentation process is finished when all particles have settled and the formation of the sediment is completed. Nevertheless, the analysis can be stopped when the last of the particles that will migrate due to gravity have passed through the measurement zone [see Figure 1 a)]. In general, measurements can be stopped when photometric signals have reached a stable value, which is associated with the particle-free supernatant.



a) Homogenous mixing state and corresponding concentration profile at $t = 0$

b) Formation of 4 zones and corresponding concentration profile at t_{observ}

c) Evolution of the photometric concentration at the measurement position

Key

X	photometric concentration
Y	position
h_{sed}	sedimentation distance
t	time
t_{observ}	time point of observation
1	zone 1 = particle-free supernatant
2	zone 2 = depleted dispersion phase (due to loss of coarse particles)
3	zone 3 = original dispersion phase
4	zone 4 = sediment
a	Sedimentation cell.
b	Light beam, with fixed or varying position.
c	Measurement zone.
d	Photometric sensor.

The grey area between the light beam and the sensor depicts the measurement zone.

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Figure 1 — Phase separation due to sedimentation for a bi-disperse sample with positive density contrast and its monitoring via light transmission

Within the scope of this document, the techniques should only be applied to dilute samples, in which particles settle or rise independently from each other (i.e. no hydrodynamic hindrance, see Reference [2]

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and ISO 13317-1). This condition offers a chance to quantify individual particle properties, such as size. Yet in principle, photosedimentation techniques also allow the characterization of concentrated samples, e.g. for the purpose of dispersion separation or stability analysis (see Annex E).

5.2 Primary and derived measurement results

The primary measurement data of photosedimentometer are optical signals from the sample contained in a transparent measurement cell (e.g. cuvette). Such signals are either the intensities of transmitted light or scattered light, which are frequently presented as normalized values (e.g. transmittance, relative scattering intensity). In general, they may be registered either as functions of time at a (multiple) vertical cell position(s) or at vertical position(s) at a fixed time(s). Several types of original measurement results are possible:

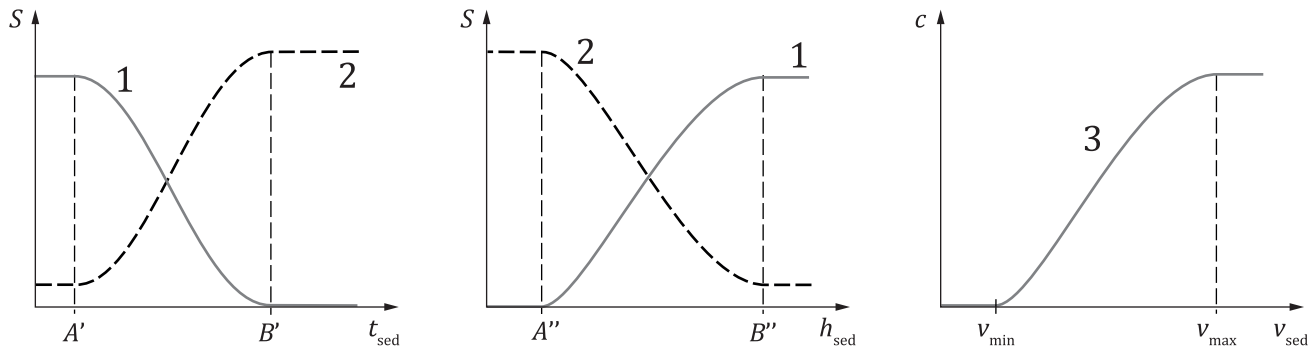
— time course of the optical signal at a constant vertical position of the sedimentation cell;

— time courses of the optical signal at few vertical positions of the sedimentation cell;

— time courses of the optical signal at different positions obtained by vertical scanning along the sedimentation cell (allows reconstruction of vertical profiles if scanning rate is faster than rate of phase separation);

— time-resolved vertical profiles of the optical signal along the measurement cell (i.e. simultaneous time courses of the optical signals at multiple, finely resolved vertical positions).

Hence, there are two principal types of original measurement results: time courses for a specified position or vertical profiles for a specified time [see Figure 2 a) and b)].



a) Primary measurement: Optical signal vs. sedimentation time

b) Primary measurement: Optical signal along vertical position

c) Derived result: Particle concentration vs. sedimentation velocity

Key

1	scattering signal
2	transmission signal
3	cumulated particle concentration
A'	time, at which the fastest particles have completely passed the vertical measurement position
B'	time, at which the slowest particles have left the zone above ($\Delta\rho > 0$) or below ($\Delta\rho < 0$) the measurement position
A''	distance in the direction of sedimentation, below which the liquid is completely depleted of the fastest particles
B''	distance in the direction of sedimentation, below which the liquid is free of particles
c	particle concentration
h_{sed}	sedimentation distance
S	signal

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t_{sed} sedimentation time

v_{sed} sedimentation velocity

The real signal shape of the schematic curves depends on several factors, including particle size distribution, particle shape, wavelength or refractive indices.

Figure 2 — Primary and derived measurement signals of gravitational photosedimentation

The primary optical signals correlate with the particle concentration but are also affected by the optical configuration of the instrument, the optical properties of the measurement cell and the optical properties of the dispersion medium. The quantification of particle fractions, i.e. for photosedimentometer applications within this document, requires a signal transformation to get rid of the signal contributions not caused by particles (see 6.1).

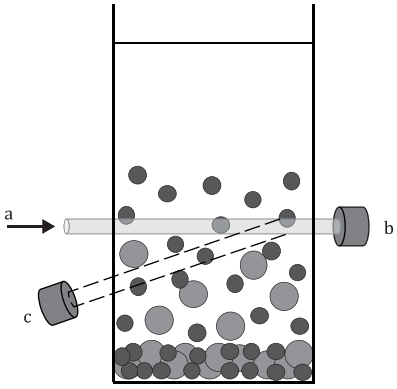
— Transmitted signal intensity is first converted into sample transmission and finally into extinction.

— Scattered intensity is converted into relative excess scattering signals or excess Rayleigh ratio.

The transformed signals are considered to be proportional to the particle concentration, which is prerequisite for the quantification of particle fractions. The evolution of signals with time or vertical position is due to the sedimentational separation and segregation of the dispersed phase as shown in Figure 1. It reflects changes in the composition of the dispersed phase and thus the quantities of particle fractions that have just left the measurement zone. Those fractions migrate with a sedimentation velocity, which is identical to the ratio of sedimentation distance to measurement time and which can be further transformed into an equivalent diameter, the Stokes diameter (see 6.1). Hence, time-curves or profiles of transmitted or scattered signals can be considered as scaled sum functions of the velocity or particle size distribution [see Figure 2 c)].

5.3 Instrumentation

Typical photosedimentometers essentially consist of a vertically aligned measurement cell (or sedimentation column), a unit for adjusting to a constant temperature in the measurement cell (e.g. water bath, contact cooling and heating), an illuminating light source or sources operating at different wavelengths, optical detection system(s) and an electronic signal processing unit.



Key

a Incident beam.

b Transmission detector.

c Scattering detector.

Figure 3 — Transmission and scattering setup

The most important feature for grouping photosedimentometer is whether they employ transmitted light[3],[4] or scattered light[5] for monitoring phase separation. The former has the advantage of a defined measurement volume, which simplifies the “extraction” of particle-induced signal contributions. On the other hand, it principally requires a certain minimum sample opacity and turbidity and allows the quantification

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of separate particle fractions, i.e. for the determination of velocity distribution or particle size distribution, only if opacity keeps below a critical threshold. Scattered light allows the monitoring of phase separation for fully transparent samples (i.e. low particle concentrations) and, in the case of backscattered (reflected) light, for opaque samples. However, the reliable quantification of separate particle fractions from scattered light requires low optical concentrations.

NOTE 1 Photosedimentometers used for the quantification of separate particle fractions, i.e. for the determination of the velocity distribution or the particle size distribution, are typically operated in transmission mode.

The optical detection system may be comprised of one (or more) detector(s) at fixed vertical position(s) or a movable detector, which scans the sample along the vertical axis. The width of the illuminating light beam(s) can be adjusted to ensure optimum signal-to-noise ratio. An alternative is spatially resolved detectors (e.g. a linear CCD array), which ideally allow the simultaneous observation of concentration changes at each vertical position (STEP Technology[2]). For this purpose, the measurement cell is illuminated with a two-dimensional beam. In practice, each type of detection system can be attributed a certain vertical resolution Δh, which affects the minimum resolution of the distribution functions (see Annex A).

Illumination in photosedimentometers employs radiation from the optical domain including visible light, UVA and NIR:

— at one, fixed wavelength (or a very narrow part of the spectrum);

— at multiple, fixed wavelengths;

— with spectral resolution (i.e. for a large number of consecutive wavelengths).

Currently, the first two are the most frequently used.

The central part of a photosedimentometer is the measurement cell, in which the liquid dispersion sample is placed for gravitational phase separation. Measurement cells for transmission measurements are typically rectangular in cross-section with the light beam perpendicularly passing two parallel windows, whereas scattering-based photosedimentometry is frequently performed on round cuvettes. The most important characteristics are the height and the optical path (diameter) of the measurement cell. The height limits the sedimentation distance and thus the maximum sedimentation velocity, which can be accessed for a given time resolution. The optical path affects the overall turbidity of the sample and thus restricts the particle concentration, at which the measurements must be run. In some cases, instruments employ different cells (cell heights, path lengths, and material) to increase flexibility regarding measurable samples. In addition, it also sets an upper limit for particle size because the photometric principle of particle quantification relies on a large number of particles in the measurement zone, otherwise signals would reflect stochastic number variations of illuminated particles. On the other hand, to avoid hydrodynamic wall effects, the width should be at least 10 times larger than the coarsest expected particles.[2],[6]

NOTE 2 For the sake of illustration, a transmission configuration can be assumed with a measurement volume of 10 mm path × 2 mm (width of beam) × 1 mm (height of beam) intended to analyse a sample of 10 µm spheres at a volume fraction of 0,1 %. This coincides with approximately 38 200 illuminated particles and, by means of Poisson statistics, the relative standard deviation of this number can be estimated as 0,5 %. For 200 nm and 100 µm nm spheres, the same volume fraction yields an average number of 5×10^9 and 38 illuminated particles, respectively. The corresponding relative standard deviation amounts to 0,01 % and 16 %, respectively.

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