International
Standard

ISO 13317-5

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

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

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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.

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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.

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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]

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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

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[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]

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

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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

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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.

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 $r = 3$

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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)	$\mathrm{m}^{0,5}\cdot\mathrm{s}^{0,5}$
C_{ext}	extinction cross section	
c_M	concentration with respect to extensive property M	varying
D_p	particle diffusion coefficient	$\mathrm{m}^2\cdot\mathrm{s}^{-1}$
E	extinction	
g	gravitational acceleration	$\mathrm{m}\cdot\mathrm{s}^{-2}$
h_sed	sedimentation distance	m
I	light intensity	
k	coverage factor	dimensionless
K_ext	extinction efficiency	
$\mathrm{k_B}$	Boltzmann constant	$\mathrm{J}\cdot\mathrm{K}^{-1}$
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

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In addition, the following subindices are frequently employed.

app	apparent
c	combined
cr	critical
ext	extinction
int	intensity

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

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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