
**Determination of particle density by
sedimentation methods —**

**Part 1:
Isopycnic interpolation approach**

*Détermination de la densité de particules par méthodes de
sédimentation —*

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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

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A list of all parts in the ISO 18747 series can be found on the ISO website.

Introduction

Dispersions are widely used in industry and everyday life. There is a need to understand the density of dispersed particles or droplets, e.g. for physico-chemical calculations like kinematic viscosity of dispersions (ISO 3105), determination of particle size distribution by separation techniques[4][5][7], characterization of core/shell or capsule-like particles, determination of particle compressibility[10] or optimization of dispersion stability by density matching[11].

The density of a body is its mass divided by its volume. This is straightforward for the mass of a larger body or particle. However, experimental determination of the volume of a macroscopic body is difficult. The geometrical volume (length, width and thickness) and the volume relevant for the determination of density can differ due to surface irregularities, fractures, fissures and open and closed pores or the measuring techniques employed.

Density determination of micro-particles, in particular nanoparticles dispersed in a liquid, raises issues, not only for the determination of mass and volume due to the small size but also, and mainly, because of the boundary between the liquid and the particle, which is fuzzy. Molecules in the continuous phase are partially immobilized at the surface. Physico-chemical properties (e.g. viscosity, ion concentration) in the fuzzy coat differ from bulk. This is especially important for small microparticles and nanoparticles which are dispersed in a polymer or biological fluid[12]. The so-called corona can be interpreted as an integral part of the particle and increases the effective/apparent volume compared to the space occupied by the dry material. The thickness of this layer ranges between a few to tens of nanometres and the effective/apparent volume deviates increasingly from the “geometrical” volume, if the particles become smaller. As a consequence, density determination by traditional methods is affected.

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Determination of particle density by sedimentation methods —

Part 1: Isopycnic interpolation approach

1 Scope

This document specifies a method for the determination of the density of solid particles or liquid droplets (below referred to generically as “particles”) dispersed in a liquid. The method is based on the fact that a particle wholly immersed in fluid experiences buoyancy equal to the weight of the fluid displaced by this particle (Archimedean principle), and if its mass force matches the buoyant force, it stops gravitational or centrifugal settling/creaming and the particle remains suspended. This implies that the density of the particle equals the density of the liquid. In this document, particle density determination is conducted by analysing the direction of the migration movement of particles dispersed in liquids with densities that are lower and higher than particle density. All particles are of the same material composition.

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 14887:2000, *Sample preparation — Dispersing procedures for powders in liquids*

3 Terms and definitions

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

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

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

3.1 dynamic viscosity

measure of the resistance of a fluid which is being deformed by shear stress

Note 1 to entry: Dynamic viscosity is calculated by shear stress divided by shear rate and determines the dynamics of an incompressible Newtonian fluid.

3.2 migration

directed particle movement (sedimentation or creaming/flotation) due to acting gravitational or centrifugal fields

Note 1 to entry: Sedimentation occurs when density of particles is larger than that of liquid density. Creaming/flotation occurs when density of particles is smaller than that of liquid density. In these two processes, particles move in opposite directions.

3.3 migration velocity

absolute value of sedimentation or creaming/flotation terminal velocity

Note 1 to entry: Velocity of creaming/flotation is indicated by a negative sign.

3.4 true particle density

ratio of particle mass to particle volume excluding all pores, closed or open, and surface fissures

3.5 skeletal density

ratio of the mass of discrete pieces of solid material to the sum of the volumes of the solid material in the pieces and closed (or blind) pores within the pieces

[SOURCE: ASTM D3766]

3.6 buoyant density

ratio of particle mass to particle volume including filled or closed pores as well as adjacent layers of liquid or other coating materials

4 Symbols

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

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Quantity	Symbol	Unit	Derivative unit
Acceleration	a	m/s ²	
Angular velocity	ω	rad/s	
Dynamic viscosity	η	Pa·s	mPa·s
Force due to buoyancy	F_B	N	
Force due to gravity	F_G	N	
Liquid density	ρ_L	kg/m ³	
Particle density	ρ_P	kg/m ³	
Radius	r	m	mm
Rotational frequency	n	s ⁻¹	min ⁻¹
Standard acceleration due to gravity	g	m/s ²	
Temperature	ϑ	°C	
Time	t	s	
Velocity	v	m/s	
Velocity measurand	y		
Volume	V	m ³	
Wavelength	λ	m	nm

5 Basic principle of method

The Archimedean principle states that a particle wholly immersed in liquid experiences buoyancy equal to the weight of the fluid displaced by this particle. The balance of the weight forces of a particle due to gravity, F_G , and of buoyancy force, F_B , determines whether its net gravitational motion is upward, downward, or neither [Formula (1)].

$$F_G = \rho_P \cdot V \cdot g = F_B = \rho_L \cdot V \cdot g \tag{1}$$

where

- ρ_P is the density of the particle;
- V is the particle volume;
- g is the gravitational acceleration;
- ρ_L is the density of the liquid.

A particle stops migration if forces F_G and F_B are equal, and therefore the density of the particle shall equal the density of the liquid ($\rho_P - \rho_L = 0$). These considerations are applicable, independent of size or shape of the particle, as well as independent on dynamic viscosity of continuous phase. In the case of multicomponent dispersions, all particle species stay suspended only when the density of all particles is the same as the liquid density.

[Formula \(1\)](#) and the above considerations also hold for dispersed particles in a centrifugal field, where g shall be replaced by a centrifugal acceleration a [[Formula \(2\)](#)].

$$a = \omega^2 \cdot r = (2 \cdot \pi \cdot n)^2 \cdot r \quad (2)$$

where

- ω is the angular velocity of the rotor;
- r is the distance of the particle under consideration from the centre of revolution;
- n is the number of rotations per seconds of the rotor.

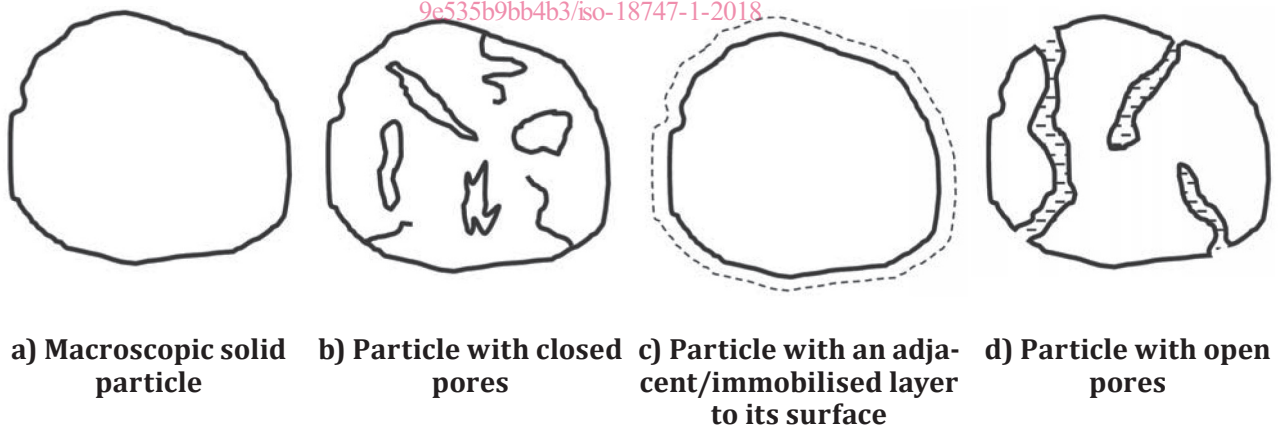


Figure 1 — General structures of particles regarding density determination [21]

According to [Formula \(1\)](#), density corresponds to the volume of displaced liquid. This equals the geometrical volume of a macroscopic solid particle [[Figure 1 a\)](#)]. It follows that determined density equals true solid particle density, which corresponds to the true material density. If the particle is made of different materials, true density reflects the (material) density and volume/mass fraction of each material.

In case of a particle with closed pores [[Figure 1 b\)](#)] or pores not filled with liquid of continuous phase, as well as with a surface layer [[Figure 1 c\)](#)], [Formula \(1\)](#) determines the buoyant density, ρ_B (also called

effective, average, or apparent density), of the particle of total volume, V_{tot} , which can be calculated according to [Formula \(3\)](#):

$$\rho_B = \frac{\rho_1 \cdot V_1 + \rho_2 \cdot V_2}{V_{\text{tot}}} \quad (3)$$

where

ρ_1 is the skeletal density;

V_1 is the volume of the skeleton;

ρ_2 is the density of the material of closed and open not filled pores;

V_2 is the volume of the pores;

V_{tot} is the total volume.

[Figure 1 b](#)) shows particles with closed pores. The same applies to particles coated with a layer of a different material of a volume of V_2 [[Figure 1 c](#)]. The term “coated” is used in a generic way. It can refer to a shell of different materials (e.g. silica coated magnetic nanoparticles), a particle brushed by polymers or macromolecules or even an immobilised (unstirred) layer of liquid molecules. The layer itself can be porous or exhibit material gradients. [Formula \(3\)](#) is based on the assumption that the composition of volume V_2 does not change when dispersing the particles into different test liquids (see [Clause 6](#)). Volume V_2 is not mixed or exchanged with the continuous phase or at least only with a time constant larger than the experimental measuring time. The term “unstirred” also reflects the fact that, in case the particle exhibits any movement (e.g. due to electrical or gravity fields), then volume V_2 shall be treated as an “integral part” of the particle, which moves together with the main particle. It is obvious that the density determined according to [Formula \(3\)](#) deviates more and more from the true particle density if particles become smaller (e.g. nanoparticles).

[Figure 1 d](#)) shows a particle that has open pores, whose content can be freely exchanged with the continuous phase. In other words, density determination applies to particles whose pores are filled with the continuous phase. In this case, skeletal density is determined.

This document focuses on buoyant density, which coincides with the true (material) density [[Figure 1 a](#)]), with apparent density [[Figure 1 b](#)] and [[Figure 1 c](#)]) and with skeletal density [[Figure 1 d](#)]). The above four cases are separately discussed for clarity, but particles which belong to several cases exist, e.g. particles with closed pores and open pores filled with the liquid of continuous phase.

6 Measuring techniques to determine the direction of gravitational and centrifugal migration of dispersed particles

The determination of particle density, according to [Formula \(1\)](#) or [\(2\)](#), is based on the detection (measurement) of the migration direction of particles dispersed in continuous liquid phases with different densities, which are lower or higher than the expected particle density.

Any method which allows detection of the direction of particle migration (sedimentation or creaming/flotation) is appropriate. Particle migration may be driven by earth gravity or centrifugal field. Basic principles available include:

- a) monitoring the concentration change near an appropriate interface, e.g. below the meniscus (dispersion/air interphase), or above the bottom of the measuring cell, either directly or by a concentration related signal, e.g. voltage, current, light intensity, X-ray absorption, conductivity, or electro-acoustic;
- b) measuring the value of migration velocity or a directly correlating measurand of dispersed particles in the bulk of the dispersion;

c) sedimentation or creaming/flotation in a density gradient¹⁾.

Attention should be paid to temperature changes. Instruments which allow for temperature setting and control are preferable. Alternatively, measurements can be conducted within a short time. Multichannel instruments are advantageous as they increase the sample throughput, and samples are measured under similar experimental conditions. Analytical cuvette centrifugation is especially appropriate for nanoparticles and continuous phases of high viscosity.

The volume fraction of dispersed phase does not enter into the calculations in [Formulae \(1\)](#) and [\(2\)](#). Choose the final volume concentration in accordance with the specification of the analytical technique employed. High volume fraction of particles (greater than 5 %) should be avoided in that, because migration velocity decreases with increasing volume concentration due to hydrodynamic interaction (hindrance)^{[13][14][20]}, experimental determination of the migration direction can be more difficult and time-consuming.

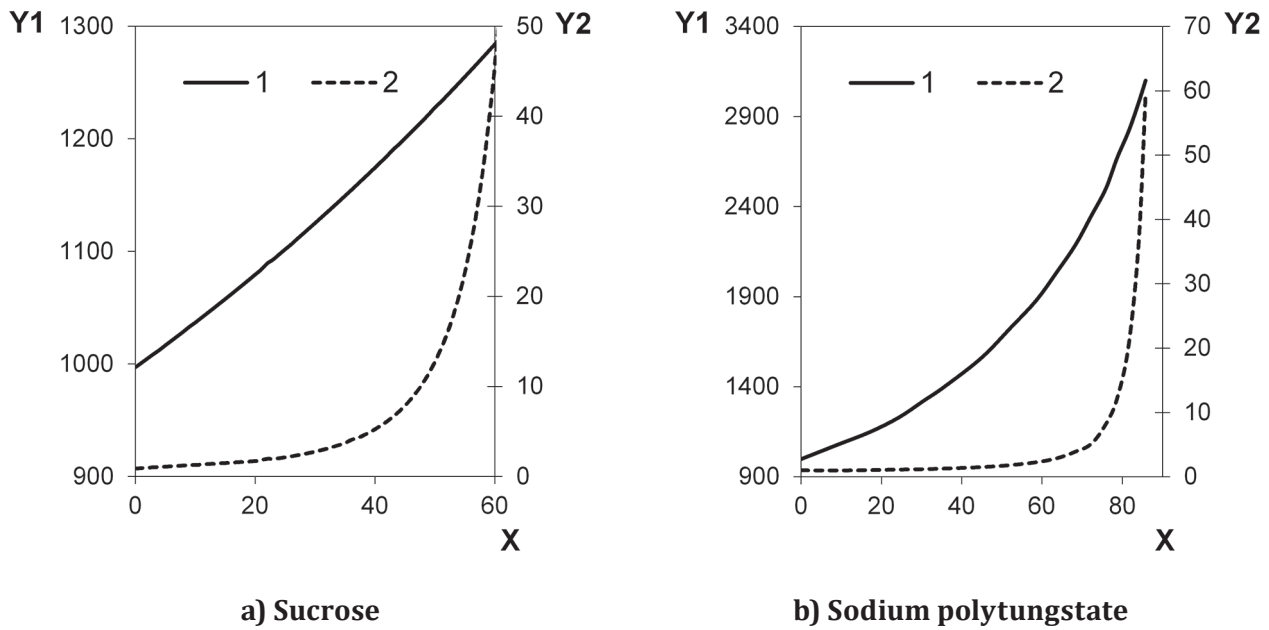
7 Preparation of samples

7.1 Solutions

Solutions which differ in density but cover the expected particle density range should be prepared. It is convenient to start with a concentrated solution and dilute with a solvent until the liquid density ρ_L is smaller than the buoyant particle density, ρ_p . Prepare two solutions as a minimum, but for more precise results, a series of five to eight test liquids where the median density corresponds to the approximate particle density is recommended. If possible, density spacing should be about equidistant. A number of suitable solutions are tabulated in the literature (e.g. References [\[15\]](#) and [\[16\]](#)). Typical examples are shown in [Figure 2](#).

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1) The buoyant density centrifugation method is not part of this document. Nevertheless, a short description is given in [Annex D](#).



Key

X mass fraction in %

Y1 density ρ in kg/m³

Y2 dynamic viscosity η in mPa·s

1 density dependence

2 viscosity dependence

NOTE See References [15] and [16] for more information.

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Figure 2 — Density and dynamic viscosity dependence on sucrose and sodium polytungstate mass fraction at $\vartheta = 25\text{ °C}$

Another approach consists of mixing two liquids of different density. Typical examples include water-ethanol-mixtures or water-glycerol mixtures. Both of them are well characterized[18][19]. Densities of these mixtures range from 789,7 kg/m³ to 998,2 kg/m³ and from 998,2 kg/m³ to 1 263,9 kg/m³ at $\vartheta = 20\text{ °C}$, respectively.

NOTE Numerical values of density as well as dynamic viscosity are functions of temperature. If the density and viscosity values are not known for a specific temperature, they can be experimentally determined according to International Standards (viscosity: ISO 3105, density: ISO 2811-3).

CAUTION — Particles, especially of organic or hydrocolloid matter, should not swell or shrink in chosen liquids due to the effects of solvation or osmotic pressure. In case of particles with open pores, liquids shall wet the pore material (contact angle > 90°) and the preparation time of dispersion shall allow fully filled pores. Furthermore, the liquid selected should not allow gelation or particle network formation.

7.2 Dispersing procedure

Disperse powders in the test liquids in accordance with the procedures specified in ISO 14887. A mild dispersing procedure is sufficient because, in contrast to other methods, e.g. particle sizing, any aggregates, agglomerates or flocks do not disturb the density measurement. Wet all particles thoroughly to avoid density underestimation due to adhering gas bubbles remaining in the test liquid.

The volume concentration of all samples should be the same and in accordance with the requirements of the measuring technique. In general, high concentrations should be avoided, as the sensitivity of migration detection will be reduced due to particle hindrance[20].